

DOCKET NO.: 244625US0

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE APPLICATION OF:
CHARLOTTE FAMY, ET AL.

SERIAL NO: 10/691,638



GROUP: 1755

EXAMINER: MARCANTONI, P.

FILED: OCTOBER 24, 2003

FOR: CEMENTITIOUS PRODUCT IN PANEL FORM AND MANUFACTURING
PROCESS

SUPPLEMENTAL DECLARATION UNDER 37 C.F.R. 1.132

COMMISSIONER FOR PATENTS
ALEXANDRIA, VIRGINIA 22313

Sir:

I, Charlotte Famy, declare and state as follows:

1. I am a named coinventor in the above-identified application.
2. I am familiar with the claims, and have read the Office Action mailed June 28, 2005,
in the above-identified application.

3. The following experiments were conducted under my supervision and/or control.
4. Hatscheck slabs were analyzed for the affect of limestone content on hydration, as
explained below:

Purpose: To evaluate the effect of the level of replacement of cement with limestone on the degree of hydration of Hatscheck slabs by silicon nuclear magnetic resonance (NMR ^{29}Si). (This analysis technique provides information about the structure of the phases containing silica in the form of tetrahedrons such as the anhydrous phases of Portland cement (C_3S /alite and C_2S /belite or hydrous phases (C-S-H ))

Four Hatscheck slabs, manufactured by Eterbras, were analyzed:

- Slab A: 20% limestone, 2-3% polyvinyl alcohol (PVA).
- Slab B: 40% limestone, 2-3% PVA
- Slab C: 60% limestone, 2-3% PVA

-Slab D: 60% limestone, 1.8 polypropylene (PP)

Table 1 gives the complete composition and the mechanical performances of these slabs:

Table 1

	% OPC	%CaCO ₃	%Cellulose (60)	% Synthetic Fiber	MOR dry	MOR wet	WM (%)
A	74.7	20	3	2.3 PVA	19.9	13.6	0.31
B	54.7	40	3	2.3 PVA	15.8	10	0.29
C	34.7	60	3	2.3 PVA	14.7	9.2	0.18
D	35.2	60	3	1.8 PP	14.0	9.0	0.20

The increase of the limestone content of the slabs from 20 to 40% does not change the shrinkage value (0.31 and 0.29%). On the other hand, when the level of limestone increases from 40 to 60%, the shrinkage decreases from 0.29 to 0.18%. It also is observed that the loss of resistance to bending (or MOR) is significant when the limestone content increases from 20 to 40%. The slabs containing 60% limestone show resistances similar to those containing 40% of same.

The added results shown in Table 1 of the Supplemental Declaration demonstrate that various synthetic fibers (e.g. PVA or PP) play the same role as a reinforcement fiber.

Specifically, both PVA and PP provide similar and satisfactory mechanical properties. Moreover, cellulose (natural fiber) has much less influence in mechanical properties as do the various synthetic fibers.

Accordingly, the data presented in this declaration illustrate that Portland cement and synthetic fibers, as claimed, gives lower shrinkage value (WM %) with a low content of Portland cement and high content of calcium carbonate (Examples C and D) relative to the cited references which describe a high Portland cement content and a low calcium carbonate content (Example A). A similar shrinkage value is reached in systems C and D irrespective of the nature of the synthetic fibers. In addition, these data are representative of the claims and

the results are surprising in that acceptable mechanical properties and low shrinkage values can be achieved.

The NMR ^{29}Si was used in order to make it possible to better understand the effect of the limestone content on the degree of hydration of the cement and the structure of the hydrates (C-S-H) formed. Table 2 presents the results. It is clearly demonstrated that the addition of limestone from 20 to 40% increases the level of hydration of the cement from 69% to 88%. This effect is explained by the increase in nucleation sites due to the presence of fine grains of limestone. The cement is activated by the limestone. The C-S-H formed in slab B are also more polymerized (longer C-S-H chain length or $\text{I}(\text{Q}^2)$) than those formed in the slab containing 20% limestone. This is in keeping with a higher level of hydration.

When the limestone content increases from 40 to 60%, the hydration level remains constant (88 and 87% respectively). Thus there is a plateau effect of limestone content on hydration level.

Knowing the quantity of anhydrous cement contained in the slabs, it is possible to determine the quantity of C-S-H formed in each slab (Table 2). A similar quantity is present in slabs A and B (51.5 and 48.1 respectively), despite a different hydration level of the cement (69 and 88%). A lesser quantity of C-S-H is present in slab C (30.2%).

Shrinkage stability, observed in slabs A and B (Table 1, 0.31 and 0.29%), is explained by a like quantity of C-S-H in these two slabs - the C-S-H being the majority compound sensitive to moisture fluctuations (shrinkage and expansion). By significantly reducing the quantity of C-S-H, the shrinkage is reduced, as is seen in slab C.

On the other hand, the preservation of resistances when the limestone content increases from 40 to 60% is provided by the microstructure of the slabs. Despite a lesser quantity of hydrates or C-S-H in slab C, because of a better spatial distribution of the hydrates and the porosity, resistance does not decline. A microstructure in which the hydrates form homogeneously in the space initially occupied by water (the outer C-S-H), filling in the micro-

and nanometric pores, offers mechanical properties superior to a microstructure favoring hydrates around grains of cement (inner C-S-H).

Table 2: % of hydration and quantity of hydrated cement in the slabs A, B and C.

%I(Q²) is the quantity of silica tetrahedrons surrounded with two silica tetrahedrons.

	% Hydration	I(Q ²)%	% OPC slabs	% hydrated OPC in slabs
A	69	33	74.7	51.5
B	88	54	54.7	48.1
C	87	57	34.7	30.2

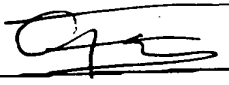
5. The undersigned declares further that all statements made herein of her own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

6. Further declarants saith not.

Customer Number

22850

Tel. (703) 413-3000
Fax. (703) 413-2220
(OSMMIN 07/05)


Signature

Date

03/02/2006